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DERWENT-ACC-NO: 1998-537509

DERWENT-WEEK: 199846

TITLE: Nitrile compounds contg. fluorine-atom - used for preparing polymers with good storage stabilities

PRIORITY-DATA: 1997JP-0037489 (February 21, 1997)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
JP 10237130 A	September 8, 1998	N/A	007	C08F 114/18

INT-CL (IPC): C07C255/13, C07D251/24 , C08F114/18 , C08F290/06 , C08F299/02

ABSTRACTED-PUB-NO: JP 10237130A

BASIC-ABSTRACT:

A nitrile compound of formula:
CH2=CFCF2O(CF2O)x(CF2CF2O)y(C(X1)2CF2CF2O)z-(CF(X2)CF2O)wCF(X3)-CN is new, where X1 = H, F or Cl; X2 = H, Cl, CH3 or CF3;
X3 = H, F, Cl or CF3; x,y and z = 0 - 20, x + y + z = 20 or less. Also claimed is a polymer obtd. from novel cpd(s)., where polymer has a molecular weight of 1,000 - 1,000,000.

USE - The nitrile cpd. is used for (co)monomers. (1) is used for curing agents esp.. The (co)polymer is used for macro-electrolytes.

ADVANTAGE - The polymer with good storage-stabilities is obtd. and with high yield.

(19)日本国特許庁（ＪＰ）　(12)**公開特許公報**（Ａ）　(11)特許出願公開番号

特開平10－237130

(43)公開日　平成10年(1998)９月８日

(51)Int.Cl.[6]	識別記号	ＦＩ
Ｃ０８Ｆ 114/18		Ｃ０８Ｆ 114/18
Ｃ０７Ｃ 255/13		Ｃ０７Ｃ 255/13
Ｃ０７Ｄ 251/24		Ｃ０７Ｄ 251/24
Ｃ０８Ｆ 290/06		Ｃ０８Ｆ 290/06
299/02		299/02

審査請求　未請求　請求項の数５　ＯＬ　（全７頁）

(21)出願番号　特願平9－37489

(22)出願日　平成９年(1997)２月21日

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(54)【発明の名称】　含フッ素ニトリルおよびその重合体

(57)【要約】

【課題】　保存安定性がよくかつ重合活性が高い新規な含フッ素ニトリルを提供する。

【解決手段】　一般式：

$CH_2=CFCF_2O-(CF_2O)_x-(CF_2CF_2O)_y-(CX^1{}_2CF_2CF_2O)_z-(CFX^2CF_2O)_w-CFX^3-CN$

（式中、X^1は、水素原子、フッ素原子または塩素原子、X^2は水素原子、塩素原子、メチル基またはトリフルオロメチル基、X^3は水素原子、フッ素原子、塩素原子またはトリフルオロメチル基を表す。ｘ、ｙ、ｚおよびｗはそれぞれ独立に０～２０の数を表す。ただし、ｘ、ｙ、ｚおよびｗの和は２０を越えない。）で示される含フッ素ニトリル。このニトリルは、それ自体で重合して、または他の共重合可能な単量体と共重合して、架橋サイトを有するポリマーを与える。

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【特許請求の範囲】

【請求項１】　一般式：

$CH_2=CFCF_2O-(CF_2O)_x-(CF_2CF_2O)_y-(CX^1{}_2CF_2CF_2O)_z-(CFX^2CF_2O)_w-CFX^3-CN$

（式中、X^1は、水素原子、フッ素原子または塩素原子、X^2は水素原子、塩素原子、メチル基またはトリフルオロメチル基、X^3は水素原子、フッ素原子、塩素原子またはトリフルオロメチル基を表す。ｘ、ｙ、ｚおよびｗはそれぞれ独立に０～２０の数を表す。ただし、ｘ、ｙ、ｚおよびｗの和は２０を越えない。）で示される含フッ素ニトリル。

【請求項２】　$CH_2=CFCF_2O-CF(CF_3)-CN$、

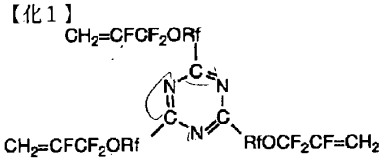
$CH_2=CFCF_2O-CF(CF_3)CF_2O-CF(CF_3)-CN$または

$CH_2=CFCF_2O-[CF(CF_3)CF_2O]_2-CF(CF_3)-CN$

である請求項１に記載の含フッ素ニトリル。

【請求項３】　一般式：

【化１】



(IV)

（式中、Ｒｆは式：$-(CF_2O)_x-(CF_2CF_2O)_y-(CX^1{}_2CF_2CF_2O)_z-(CFX^2CF_2O)_w-CFX^3-$（ここで、$X^1X^2X^3$ｘｙｚおよびｗは前記と同意義である。）で示される基を表す。）で示される含フッ素多官能トリアジン化合物。

【請求項４】　分子量が１，０００～１，０００，０００である、請求項１または２に記載の含フッ素ニトリルの重合体。

【請求項５】　分子量が１，０００～１，０００，０００＊

$CH_2=CFCF_2O-(CF_2O)_x-(CF_2CF_2O)_y-(CX^1{}_2CF_2CF_2O)_z-(CFX^2CF_2O)_w-CFX^3-CN$（Ｉ）

※る。含フッ素ニトリル（Ｉ）の好ましい例は、

$CH_2=CFCF_2O-CF(CF_3)-CN$、

$CH_2=CFCF_2O-CF(CF_3)CF_2O-CF(CF_3)-CN$および

$CH_2=CFCF_2O-[CF(CF_3)CF_2O]_2-CF(CF_3)-CN$

である。なお、各一般式を簡略に示すために、以下の説明では$-(CF_2O)_x-(CF_2CF_2O)_y-(CX^1{}_2CF_2CF_2O)_z-(CFX^2CF_2O)_w-CFX^3-$をＲｆで表す。

【０００６】本発明の含フッ素ニトリル（Ｉ）は、一般式：

(II)

★触媒（例えば、亜鉛、銅など）の存在下に、脱ＦＩすることにより容易に合成することができる。反応温度は、－２０℃～２００℃、好ましくは５０℃～１５０℃である。

2

＊であり、含フッ素ニトリルの含有量が０．１～９９モル％である、請求項１または２に記載の含フッ素ニトリルと他の共重合可能な単量体との共重合体。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は、含フッ素ニトリルおよびその重合体に関し、更に詳しくは、ビニル基、エーテル基およびシアン基を有する含フッ素有機化合物およびその重合体に関する。

【０００２】

【従来の技術】官能基（例えばビニル基、シアン基など）を有するモノマーを重合することによりポリマー中に官能基を導入し、架橋サイトなどの反応活性点として利用することは知られている。例えば米国特許第３５４６１８６号、同第３９３３７６７号、同第４２８１０９２号などでは、シアン基含有パーフルオロビニルエーテルを共重合させてパーフルオロポリマーとし、シアン基を介して三量化させてパーフロロエラストマー架橋物を得ている。また、米国特許第３８５２３２６号、同第４０３１１２４号などには、この種のモノマーの合成方法が記されている。

【０００３】しかし、上記先行特許に記載されたパーフルオロ化合物は、合成方法が複雑であり、また収率も低く、結果として高価な物となっている。また、パーフルオロビニルエーテルは酸素に対する安定性が低く、また重合反応性は高くない。

【０００４】

【発明が解決しようとする課題】本発明は、比較的収率良く得られ、かつ保存安定性も良く、重合活性も高い新規な含フッ素ニトリルを提供しようとするものである。

【０００５】

【課題を課題するための手段】本発明は、一般式：

$ICH_2CF_2CF_2ORfCN$

（式中、Ｒｆは前記で定義した通りである。）で示される末端ヨウ素ニトリルを、溶媒（例えば、ジメチルホルムアミド、ジメチルスルホキシド、メチルアルコール、アセトン、メチルエチルケトン、酢酸エチルなど）中、

【0007】出発物質である末端ヨウ素ニトリル（II）＊

$$ICH_2CF_2CF_2ORfA$$

（式中、Ｒｆは前記で定義した通りである。Ａは、ＣＯＦ、ＣＯＯＨまたはＣＯＯＲ（ここで、Ｒは炭素数１～１０の有機基である。）を表す。）で示される化合物等から公知の方法（例えば、米国特許４，１３８，４２６号の記載参照）で誘導できる。

【0008】一例として$ICH_2CF_2CF_2ORfCN$の合成ルートを以下に示す。

【化２】

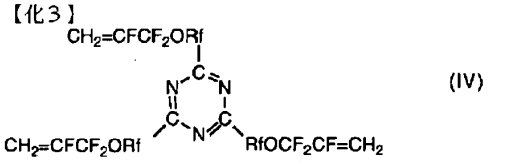
１．$ICH_2CF_2CF_2ORfCOF + CH_3OH \rightarrow ICH_2CF_2CF_2ORfCOOCH_3$

２．$ICH_2CF_2CF_2ORfCOOCH_3 + NH_3 \rightarrow ICH_2CF_2CF_2ORfCONH_2$

３．$ICH_2CF_2CF_2ORfCONH_2 \rightarrow ICH_2CF_2CF_2ORfCN$

【0009】本発明の含フッ素ニトリル（Ｉ）は、三量化すると、一般式：

【化３】



(IV)

（式中、Ｒｆは前記で定義した通りである。）で示される含フッ素多官能トリアジン化合物が得られる。このトリアジン化合物（IV）は、活性が高く、重合体の架橋剤として有用である。

【0010】含フッ素ニトリル（Ｉ）の三量化は、例えばJ.Org Chem. ３２、２３１（１９６７）に記載されている方法で合成できる。まず該ニトリルの二分子を、NH_3の存在下に反応させて$CH_2=CFCF_2ORfC(NH_2)=N-C(=NH)-RfOCF_2CF=CH_2$を得る。反応温度は特に限定されないが、－５０℃～１００℃、好ましくは－３０℃～５０℃であり、反応圧力は特に限定されないが減圧から加圧まで適用できる。反応は温度が高いほど早いが、NH_3ガスのため大気圧下で濃度を保つには温度を低くする必要がある。耐圧容器を使用すれば、例えば室温ですばやく反応さ，せることも可能である。反応溶媒は非プロトン性のもので、ニトリルと混合できるものであれば特に問題なく使用できる。たとえばテトラヒドロフラン、ジメチルホルムアミド、アセトンなどである。使用にあたっては水分を含まないよう、予め脱水するなどしておくと良い。

【0011】得られた$CH_2=CFCF_2ORfC(NH_2)=N-C(=NH)-RfOCF_2CF=CH_2$に、２倍モルの$CH_2=CFCF_2ORfCOZ$（ここで、Ｚはハロゲン原子、例えばフッ素、塩素、臭素またはヨウ素原子である。）を反応させることにより、目的とするトリアジン化合物が得られる。反応温度は特に限定されないが－５０℃～１００℃、好ましくは０℃～５０℃であり、反応圧力は特に限定されないが使用する化合物の沸※点に応じて決められるべきで、大気圧下で問題なく実行できる。反応溶媒は特に限定されないが、非プロトン性溶媒で水分を含まないものが用いられる。たとえばテトラヒドロフラン、ジメチルホルムアミド、アセトンなどである。反応にあたって受酸剤としてトリエチルアミンなどを加えると、反応が容易に進行する。

＊は、一般式：

(III)

【0012】この反応はまた、初めに該ニトリルに過剰量のNH_3を反応させ、まず$CH_2=CFCF_2ORfC(NH_2)=NH$を得てから、これに等モルの$CH_2=CFCF_2ORfCN$を反応させることにより、定量的に$CH_2=CFCF_2ORfC(NH_2)=N-C(=NH)-RfOCF_2CF=CH_2$を得、さらにこれに２倍モルの$CH_2=CFCF_2ORfCOZ$を反応させることによっても実施することができる。$CH_2=CFCF_2ORfC(NH_2)=NH$を得る反応条件は、$NH_3$を過剰に使用する以外は上記の反応と同様である。

【0013】$CH_2=CFCF_2ORfC(NH_2)=NH$と$CH_2=CFCF_2ORfCN$の反応条件は、特に限定されず、反応温度は、－５０℃～１００℃、好ましくは０℃～５０℃である。圧力についても制限はない。溶媒は特に限定されないが、非プロトン性溶媒で水分を含まないものが用いられる。たとえばテトラヒドロフラン、ジメチルホルムアミド、アセトンなどである。$CH_2=CFCF_2ORfC(NH_2)=N-C(=NH)-RfOCF_2CF=CH_2$と$CH_2=CFCF_2ORfCOB$の反応は、Ｒｆ中のｘ、ｙ、ｚおよびｗの異なる組み合わせについても同様に行うことができる。この方法により、ニトリルの任意の組み合わせでトリアジン環を得ることができる。

【0014】本発明の含フッ素ニトリル（Ｉ）は、その１種を単独重合することも、またはその２種以上を共重合することもでき、さらには、他の共重合可能な単量体と共重合することもできる。他の共重合可能な単量体としては、テトラフルオロエチレン、フッ化ビニリデン、ヘキサフルオロプロペン、パーフルオロメチルビニルエーテルやパーフルオロプロピルビニルエーテルなどのパーフルオロアルキルビニルエーテル、ヘキサフルオロイソブテン、トリフルオロエチレン、フッ化ビニル、クロロトリフルオロエチレン、エチレン、プロピレン、ブタジエン、アルキルビニルエーテルなどが例示できる。

【0015】本発明の含フッ素ニトリル（Ｉ）の重合は、一般的な重合方法により、一般的な重合条件で行うことができる。好ましくは、ラジカル開始源を用いたラジカル重合（塊状重合、溶液重合、乳化重合等）が採用される。ラジカル重合における反応条件は、特に制限されないが、０～１００℃の温度、大気圧、７６cmHg程度までの減圧または１００Kg／cm^2Ｇ程度までの加圧から選ばれる圧力を含む。

【００１６】本発明の含フッ素ニトリルは、単独重合用または共重合用のモノマーとして用いてフッ素およびシアン基を重合体に導入することができる。また、シアン基を利用してトリアジン架橋、カルボン酸等への誘導等により、ポリマーへ官能基を有利に導入することができる。シアン基のトリアジン架橋は、高性能、耐熱性のフッ素ゴムの架橋として利用でき、一方単独重合体あるいは共重合体のシアン基をカルボン酸とすることにより、重合体を高分子電解質とし、電池用電解質などとして利用することも可能である。また、イオン架橋も可能である。主鎖の脱HF反応を利用してシアン基を側鎖に持つ架橋ポリマー(ゴム)を得ることもできる。さらにシアン基をカルボン酸とした重合体は、イオン交換樹脂、高吸水高分子等として利用することも可能である。また、シアン基を利用した三量化により得られる三官能不飽和化合物は、架橋剤などとして使用できる。

【００１７】

【実施例】

実施例１

$ICH_2CF_2CF_2CF(CF_3)CN$２５ｇ、亜鉛粉末１５ｇおよびジメチルホルムアミド（ＤＭＦ）１０mlを、フラスコに入れ、これを１６０℃の油浴上で加熱した。しばらくして（約４０分後）突発的な反応が起こった。その後、混合物を７１℃で還流させて反応を１時間続けた。反応終了後、反応混合物を、常圧で単蒸留して生成物２．４３ｇ（純度９９．４％）を得た。沸点７１℃。生成物の構造を、ＩＲ、ＧＣＭＳおよびＮＭＲ(H,F)で確認したところ、$CH_2=CFCF_2CF(CF_3)CN$であった。ＩＲチャートを図１に示す。

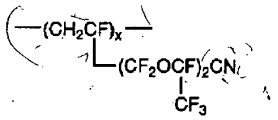
【００１８】実施例２

$ICH_2CF_2[CF_2OCF(CF_3)]_2CN$５０ｇ、亜鉛粉末２０ｇおよびＤＭＦ１０mlを、フラスコに入れ、これを１６０℃の油浴上で加熱した。しばらくして（約６＊０分後）突発的な反応が起こった。反応終了後、反応混合物を、常圧で留出させて７．７７ｇの留分を得た（純度７４％、未反応物１１％、ＤＭＦ１３％）。これを単蒸留で精製して純度９８．６％の生成物を得た。沸点１０８～１１２℃。生成物の構造を、ＩＲ、ＧＣＭＳ、およびＮＭＲ(H,F)で確認したところ、$CH_2=CF[CF_2OCF(CF_3)]_2CN$であった。ＩＲチャートを図２に示す。

【００１９】実施例３

$CH_2=CF[CF_2OCF(CF_3)]_2CN$（以下、「ＡＣＮ２」と略称する）５ｇ、$[H(CF_2CF_2)_3COO]_2$／$C_2F_3Cl_3$（８重量％）０．５ｇをガラス容器中で混合し、容器内雰囲気を窒素置換し、室温で撹拌したところ、混合物の粘度が上昇した。反応混合物から低沸点物を減圧留去して、無色透明粘稠なポリマー２．８２ｇを得た。ＩＲ、^1H－ＮＭＲ及び^{19}F－ＮＭＲの結果から、生成ポリマーは不飽和結合を含むポリマーであることが確認された。構造は、

【化４】



であった。収量およびＮＭＲの結果から計算したところ、数平均分子量は、約３５０００～約４８０００であった。

【００２０】実施例４～６

実施例３と同様にして、ＡＣＮ２と$CH_2=CF_2$、$CF_2=CF_2$または$CF_2=CF_2$／$CF_3CF_2CF_2O[CF(CF_3)CF_2O]_2CF=CF_2$（φＶＥ）との共重合を行った。結果を、表１に示す。

【００２１】

【表１】

	モノマー等	仕込量	収量	Mn(収量)	Mn(NMR)	組成(%)(NMR)
実施例４	ACN2 $CH_2=CF_2$ DHP	10g 9Kg/cm²G 1g	6.15g	53,000	43,200	ACN2: 76.9 $CH_2=CF_2$:23.1
実施例５	ACN2 $CF_2=CF_2$ DHP	10g 8Kg/cm²G 1g	6.52g	56,900	1)	ACN2: 78.8 $CF_2=CF_2$:21.2
実施例６	ACN2 φVE[2)] $CF_2=CF_2$ DHP[3)]	1g 20g 5Kg/cm²G 1g	7.99g	68,900	72,300	ACN2: 4.2 φVE: 21.5 $CF_2=CF_2$:74.3

注：1）ＮＭＲでは末端基による吸収が小さく、分子量は計算できなかった。

2）φＶＥ：$CF_3CF_2CF_2O[CF(CF_3)CF_2O]_2CF=CF_2$

3）ＤＨＰ：$\{H(CF_2CF_2)_3COO-\}_2$ 8Wt%／トリクロロトリフルオロエタン

【0022】実施例7
内容積3Lのオートクレーブに、純水1225g、$CF_3CF_2CF_2OCF(CF_3)CF_2OCF(CF_3)COONH_4$ 122.5g、NaCl 4g、Na_2SO_3 2.155gおよび$CF_3CF_2CF_2O[CF(CF_3)CF_2O]_2CF=CF_2$ 367.5gを仕込み、オートクレーブ内雰囲気をまず窒素で、次いで$CF_2=CF_2$で置換し、15℃で$CF_2=CF_2$で内圧を2.5Kg/cm²Gとし、その後、過硫酸アンモニウム24.5mgを純水10mlに溶解した溶液を仕込んだ。重合の進行につれて圧力が2.5Kg/cm²Gに低下したところで$ICF_2CF_2CF_2CF_2$ 211.57gを仕込んだ。さらに圧力が1.5Kg/cm²Gになったところで$CF_2=CF_2$で圧力を2.5Kg/cm²Gに戻した。

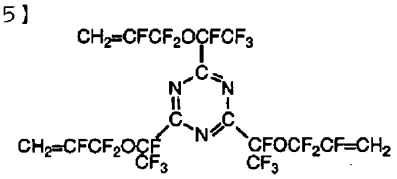
【0023】同様にして、重合圧力が1.5kg/cm²Gまで低下したところで再度$CF_2=CF_2$をボンベから仕込み、圧力を2.5kg/cm²Gに戻した。この時、ボンベの重量減少を仕込んだ$CF_2=CF_2$の重量とする。この手順を繰り返し、さらに重合が進んで$CF_2=CF_2$を合計50g仕込んだところでACN2を2.79g仕込んだ。ACN2を仕込むと重合速度が極端に遅くなったが、しばらくする内に重合速度が回復した。さらに重合を進めて、$CF_2=CF_2$を合計100g仕込んだところでもう一度ACN2を2.79g仕込んだ。前回と同様に重合速度の低下が認められたが同じように回復した。合計140gの$CF_2=CF_2$を追加したところで重合を停止した。残存ガスモノマーをブローして1892.2gの分散体を得た。固形分含量は20.87重量%であった。重合時間は合計154分であった。得られた分散体に塩酸を加えて凝析し、アセトンで洗浄し、120℃で真空乾燥して白色のゴム状ポリマーを得た。このポリマーの組成は$CF_2=CF_2$／φVE／ACN2＝76.4／22.8／0.8（モル比）であった。また、ムーニー粘度はML_{1+10}（100℃）＝41.5であった。

【0024】実施例8
ACN2の追加をさらに$CF_2=CF_2$の合計仕込量が25gおよび75gの時にも行った以外は実施例7と同様にして、ゴム状ポリマーを得た。実施例7および8で得たポリマーを薄膜にしてIRを測定したところ、どちらも－CNに基づく2250cm^{-1}付近の吸収が認められた。このポリマーの組成は$CF_2=CF_2$／φVE／ACN2＝76.1／22.7／1.2（モル比）であった。また、ムーニー粘度はML_{1+10}（100℃）＝40であった。

【0025】実施例9
実施例8で得たポリマー100重量部に3重量部のテトラフェニルスズを3インチゴムロールで混練りし、230℃で1時間プレス加硫したところ、無色透明の薄膜加硫ゴムが得られた。この薄膜のIRでは－CNに基づく吸収は消え、トリアジン環に基づく1550cm^{-1}の吸収が認められた。

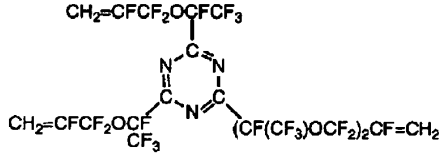
【0026】実施例10
$CH_2=CFCF_2OCF(CF_3)CN$ 51.5gに－30℃でNH_3 15gを仕込み、徐々に室温まで昇温させ過剰のNH_3を大気圧で開放し、さらに一夜反応させ、減圧蒸留によって$CH_2=CFCF_2OCF(CF_3)C(NH)=NH$を得た。（構造は、IR及びNMRにより確認した。）
得られた$CH_2=CFCF_2OCF(CF_3)C(NH)=NH$ 25.4gに等モルの$CH_2=CFCF_2OCF(CF_3)CN$ 23.7gを反応させ、$CH_2=CFCF_2OCF(CF_3)C(=NH)-N-C(NH_2)CF(CF_3)OCF_2CF=CH_2$を得た。（構造は、IR及びNMRにより確認した。）
この$CH_2=CFCF_2OCF(CF_3)C(=NH)-N-C(NH_2)CF(CF_3)OCF_2CF=CH_2$ 4.91gに2倍モルの$CH_2=CFCF_2OCF(CF_3)COF$ 5.16gを添加したところ、穏やかな発熱を認めた。そして白色固体が析出したが、しばらくすると、無色透明となった。この時点での反応混合物のIRからトリアジンの生成が認められたが、未反応原料が残存していることも確認された。これにトリエチルアミン3.03gを加えると発熱的に反応し、液は黄褐色に着色し、IR（図3）からは、原料は完全に反応して下記式のトリアジンが生成したことが認められた。

【0027】
【化5】



【0028】実施例11
実施例10と同様にして、$CH_2=CFCF_2OCF(CF_3)C(NH)=NH$に等モルのACN2を反応させ、$CH_2=CFCF_2OCF(CF_3)C(=NH)-N-C(NH_2)[CF(CF_3)OCF_2]_2CF=CH_2$を得た。これに2倍モルの$CH_2=CFCF_2OCF(CF_3)COF$およびトリエチルアミンを添加して下記化合物を得た。

【0029】
【化6】



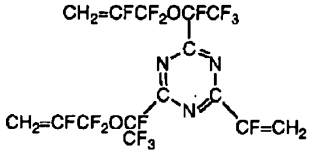
実施例12
実施例10と同様にして得た$CH_2=CFCF_2OCF$

9

$(CF_3)C(=NH)-N-C(NH_2)CF(CF_3)OCF_2CF=CH_2$に2倍モルの$CH_2=CFCOF$とトリエチルアミンを添加して、下記化合物を得た。

【0030】

【化7】



【0031】実施例13

実施例10で得たトリアジン$[CH_2=CFCF_2OCF(CF_3)CN]_3$1.0gに有機パーオキサイド（パーヘキサー2,5－B。日本油脂株式会社製）0.1gを加え、油浴上で160℃に加熱したところ、急激に反応して硬い樹脂状物が得られた。

【0032】実施例14

実施例13と同様にして、実施例10で得たトリアジン0.5gとトリアリルイソシアネート（TAIC）0.5gとを混合し、これにパーヘキサー2,5－B0.1gを加え、油浴上で160℃に加熱したところ、急激に反応し硬い樹脂状物が得られた。

【0033】実施例15および比較例1

両末端にヨウ素を有するテトラフルオロエチレンと$CF_3CF_2CF_2O[CF(CF_3)CF_2O]_2CF=CF_2$の共重合体ゴム（テトラフルオロエチレン含有量77モル%。共重合体のムーニー粘度$ML_{(1+100)}$（100℃）＝26）100重量部にSRF（カーボンフィラー）7重量部、MTC（カーボンフィラー）8重量部、実施例

10

1で得たトリアジン$CH_2=CFCF_2OCF(CF_3)CN$8.5重量部およびパーヘキサー2,5－B2部を3インチゴムロールで混練りし、JSR CURELASTOMETER IIFを用い、160℃で加硫曲線を測定した。比較として$CH_2=CFCF_2OCF(CF_3)CN$のかわりにTAIC3重量部配合する以外は上記と同様に成分を混練りした。

【0034】混練物をそれぞれ、160℃で10分間プレス加硫し、厚さ2㎜のシートを得、200℃で4時間二次加硫した。加硫シートの常態物性（破断時強度および伸び）を、ORIENTEC TENSILONを用い、JIS K 6301に準じて測定した。同様に製造した加硫シートを250℃のオーブンで70時間熱老化させた後、老化物性を測定した。結果を表2に示す。

【0035】

【表2】

	実施例15	比較例1
常態物性		
破断時強度(kgf/cm²)	86	120
破断時伸び（%）	136	100
老化物性		
破断時強度(kgf/cm²)（変化率）	64（−26%）	79（−34%）
破断伸び（%）（変化率）	170（+26%）	205（+105%）

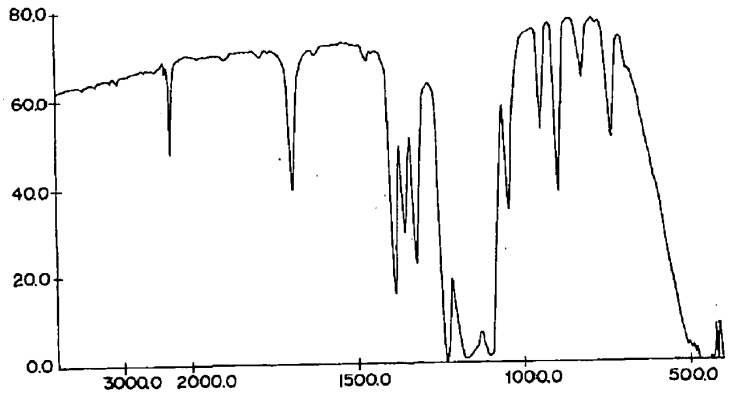
【図面の簡単な説明】

【図1】　実施例1で得た生成物のIRチャート。

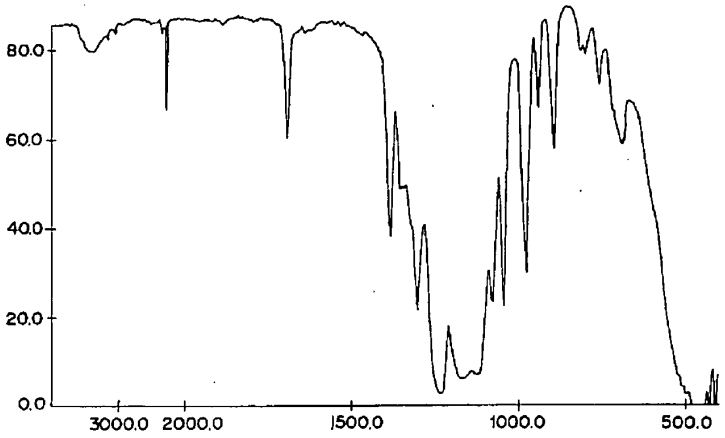
【図2】　実施例2で得た生成物のIRチャート。

【図3】　実施例10で得た生成物のIRチャート。

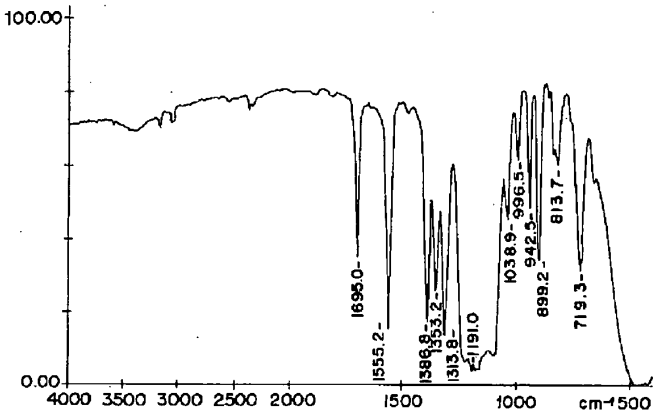
【図1】



【図2】


80.0
60.0
40.0
20.0
0.0
3000.0
2000.0
1500.0
1000.0
500.0

【図3】

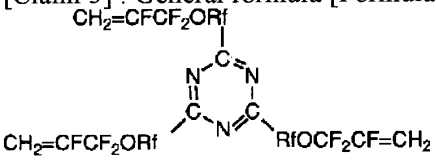

100.00
0.00
1695.0
1555.2
1386.8
1353.2
1313.8
1191.0
1038.9
996.5
942.5
899.2
813.7
719.3
4000 3500 3000 2500 2000
1500
1000
cm-1
500

* NOTICES *

1. This document has been translated by computer.So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3.In the drawings, any words are not translated.

CLAIMS

[Claim(s)]
[Claim 1] General formula :CH2=CFCF2O-(CF2O) x-(CF2CF2O) y-(CX12CF2CF2O) z-(CFX2CF2O) w-CFX3- CN (in the inside of a formula, and X1, a hydrogen atom, a fluorine atom or a chlorine atom, and X2 express a hydrogen atom, a chlorine atom, a methyl group, or a trifluoromethyl machine, and X3 expresses a hydrogen atom, a fluorine atom, x, and y, z and w express the number of 0-20 independently, respectively. However, the sum of x, and y, z and w does not exceed 20. Fluorine-containing nitril shown.
[Claim 2] Fluorine-containing nitril according to claim 1 which is CH2=CFCF2 O-CF(CF3)-CN, CH2=CFCF2 O-CF(CF3) CF20-CF(CF3)-CN, or CH2=CFCF2O-[CF(CF3) CF20]2-CF(CF3)-CN.
[Claim 3] : General formula [Formula 1]



(IV)

(-- Rf expresses among a formula the basis shown by formula:-(CF2O)x-(CF2CF2O) y-(CX12CF2CF2O) z-(CFX2CF2O) w-CFX3- (here -- it is -- X -- 1X2X3 -- xyz and w are the above and this meaning) Fluorine-containing polyfunctional triazine compound shown by).
[Claim 4] The polymer of the fluorine-containing nitril according to claim 1 or 2 whose molecular weight is 1,000-1,000,000.
[Claim 5] A copolymer with the monomer [molecular weight is 1,000-1,000,000 and] which the content of fluorine-containing nitril copolymerizes [the fluorine-containing nitril according to claim 1 or 2 it is / nitril / 0.1-99-mol % and / other].

[Translation done.]

DETAILED DESCRIPTION

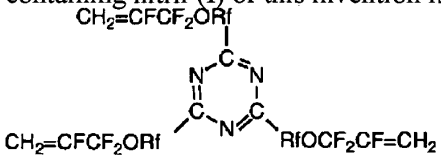
[Detailed Description of the Invention]
[0001]
[The technical field to which invention belongs] this invention relates to the fluorine-containing organic compound which has a vinyl group, an ether machine, and a cyano basis, and its polymer in more detail about fluorine-containing nitril and its polymer.
[0002]
[Description of the Prior Art] By carrying out the polymerization of the monomer which has functional groups (for example, a vinyl group, a cyano basis, etc.), a functional group is introduced into polymer and using as the reaction active spots, such as a bridge formation site, is known. For example, in U.S. Pat. No. 3546186, 3933767, and 4281092, copolymerization of the cyano basis content perfluoro vinyl ether was carried out, it considered as perfluoro polymer, 3 ****s was carried out through the cyano basis, and the perphloro elastomer bridge formation object has been obtained. Moreover, the synthetic method of this kind of monomer is describing at U.S. Pat. No. 3852326 and 4031124.
[0003] However, the perfluoro compound indicated by the above-mentioned precedence patent has a synthetic complicated method, and yield's is low, and it has become an object expensive as a result. Moreover, perfluoro vinyl ether has the low stability over oxygen, and polymerization reactivity is not high.
[0004]
[Problem(s) to be Solved by the Invention] this invention is obtained with comparatively sufficient yield, and that of preservation stability is good, and tends to offer new fluorine-containing nitril also with high polymerization activity.
[0005]
[The means for carrying out the technical problem of the technical problem] this invention is a general formula (I) (among a formula). : CH2=CFCF2O-(CF2O)x-(CF2CF2O) y-(CX12CF2CF2O) z-(CFX2CF2O) w-CFX3-CN In X1, a hydrogen atom, a fluorine atom or a chlorine atom, and X2 express a hydrogen atom, a chlorine atom, a methyl group, or a trifluoromethyl machine, and X3 expresses a hydrogen atom, a fluorine atom, a chlorine atom, or a trifluoromethyl machine. x, and y, z and w express the number of 0-20 independently, respectively. However, the sum of x, and y, z and w does not exceed 20. The fluorine-containing nitril shown, the polymer of this fluorine-containing nitril whose molecular weight is 1,000-1,000,000, and molecular weight are 1,000-1,000,000, and the content of fluorine-containing nitril offers 0.1-99-mol the copolymer of this fluorine-containing nitril it is [nitril] %, and the monomer in which other copolymerization is possible. The desirable examples of fluorine-containing nitril (I) are CH2=CFCF2 O-CF(CF3)-CN, CH2=CFCF2 O-CF(CF3) CF20-CF(CF3)-CN, and CH2=CFCF2O-[CF(CF3) CF20]2-CF(CF3)-CN. In addition, in order to show each general formula simple, the following explanation expresses -(CF2O) x-(CF2CF2O) y-(CX12CF2CF2O) z-(CFX2CF2O) w-CFX3- by Rf.
[0006] The fluorine-containing nitril (I) of this invention is a general formula. : ICH2CF2CF2ORfCN (II)

(-- Rf is as having given the definition above among a formula The end iodine nitril shown by) is easily compoundable by carrying out ** FI under existence (for example, zinc, copper, etc.) of a catalyst among solvents (for example, a dimethylformamide, dimethyl sulfoxide, methyl alcohol, an acetone, a methyl ethyl ketone, ethyl acetate, etc.). -20 degrees C - 200 degrees C of reaction temperature are 50 degrees C - 150 degrees C preferably.

[0007] The end iodine nitril (II) which is a starting material is a general formula (III) (Rf is as having given the definition above among a formula.). : ICH2CF2CF2ORfA A expresses COF, COOH, or COOR (it is here and R is the organic machine of carbon numbers 1-10.). It can guide from the compound shown by the well-known method (for example, refer to the publication of U.S. JP,4,138,426,B).

[0008] The synthetic root of ICH2CF2CF2ORfCN is shown below as an example.

[Formula 2] 1. ICH2CF2CF2ORfCOF+CH3OH -> ICH2CF2CF2ORfCOOCH32.ICH2CF2CF2ORfCOOCH3+NH3 -> ICH2CF2CF2ORfCONH23.ICH2CF2CF2ORfCONH2 -> ICH2CF2CF2ORfCN [0009] if the fluorine-containing nitril (I) of this invention is quantified three times -- general formula: -- [Formula 3]



(IV)

(-- Rf is as having given the definition above among a formula The fluorine-containing polyfunctional triazine compound shown by) is obtained. This triazine compound (IV) has high activity, and it is useful as a cross linking agent of a polymer.

[0010] 3 quantification of fluorine-containing nitril (I) is compoundable by the method indicated by J.Org Chem.32,231 (1967). First, the dyad of this nitril is made to react to the bottom of existence of NH3, and CH2=CFCF2ORfC(NH2) =N-C(=NH)-RfOCF2 CF=CH2 is obtained. Although especially reaction temperature is not limited, -50 degrees C - 100 degrees C are -30 degrees C - 50 degrees C preferably, and especially reaction pressure is applicable from reduced pressure to pressurization, although not limited. Although a reaction is so early that temperature is high, for maintaining concentration under atmospheric pressure, it is necessary to make temperature low for NH3 gas. if a proof-pressure container is used -- a room temperature -- quick -- reaction ** and **** -- things are also possible A reaction solvent is the thing of non-proton nature, and if mixable with nitril, it can be used especially satisfactory. For example, they are a tetrahydrofuran, a dimethylformamide, an acetone, etc. To dehydrate beforehand is good so that moisture may not be included in use.

[0011] The triazine compound made into the purpose is obtained by making CH2=CFCF2ORfCOZ (here, Z being a halogen atom, for example, a fluorine, chlorine, a bromine, or an iodine atom.) of a double-precision mol react to obtained CH2=CFCF2ORfC(NH2) =N-C(=NH)-RfOCF2 CF=CH2. Although especially reaction temperature is not limited, -50 degrees C - 100 degrees C are 0 degree C - 50 degrees C preferably, and especially reaction pressure should be decided according to the boiling point of the compound used although not limited, and can be performed satisfactory under atmospheric pressure. Although especially a reaction solvent is not limited, what does not contain moisture by the aprotic solvent is used. For example, they are a tetrahydrofuran, a dimethylformamide, an acetone, etc. If a triethylamine etc. is added as carrier acid in a reaction, a reaction will advance easily.

[0012] After this reaction makes NH3 of an excessive amount react to this nitril first and obtains CH2=CFCF2ORfC(NH2) =NH first, by making CH2=CFCF2ORfCN of mols [this] react again CH2=CFCF2ORfC(NH2) =N-C(=NH)-RfOCF2 CF=CH2 can be obtained quantitatively, and it can carry out also by making CH2=CFCF2ORfCOZ of a double-precision mol react to this further. The reaction condition which obtains CH2=CFCF2ORfC(NH2) =NH is the same as that of the above-mentioned reaction except using NH3 superfluously.

[0013] Especially the reaction condition of CH2=CFCF2ORfC(NH2) =NH and CH2=CFCF2ORfCN is

not limited, but -50 degrees C - 100 degrees C of reaction temperature are 0 degree C - 50 degrees C preferably. There is no limit also about a pressure. Although especially a solvent is not limited, what does not contain moisture by the aprotic solvent is used. For example, they are a tetrahydrofuran, a dimethylformamide, an acetone, etc. The reaction of CH2=CFCF2ORfC(NH2) =N-C(=NH)-RfOCF2 CF=CH2 and CH2=CFCF2ORfCOB can be similarly performed about the combination from which x in Rf, and y, z and w differ. By this method, a triazine ring can be obtained in the arbitrary combination of nitril.

[0014] Homopolymerizing one of them can also copolymerize the two or more sorts, and the fluorine-containing nitril (I) of this invention can also copolymerize it with the monomer in which copolymerization of further others is possible. As a monomer in which other copolymerization is possible, perfluoroalkyl vinyl ether, such as a tetrafluoroethylene, a fluoride vinylidene, a hexafluoro propene, the perfluoro methyl vinyl ether, and perfluoro propyl vinyl ether, a hexafluoro isobutene, truffe RUORO ethylene, a fluoride vinyl, a chlorotrifluoroethylene, ethylene, a propylene, a butadiene, alkyl vinyl ether, etc. can be illustrated.

[0015] The polymerization of the fluorine-containing nitril (I) of this invention can be performed on general polymerization conditions with a general polymerization method. Preferably, the radical polymerizations (a bulk polymerization, solution polymerization, emulsion polymerization, etc.) using the source of a radical start are adopted. Although especially the reaction condition in a radical polymerization is not restricted, it contains the pressure chosen from the temperature of 0-100 degrees C, atmospheric pressure, the reduced pressure to about 76 cmHgs, or the pressurization to about 100 kg/cm2G. [0016] The fluorine-containing nitril of this invention can be used as a monomer for the object for homopolymerization, or copolymerization, and can introduce a fluorine and a cyano basis into a polymer. Moreover, a functional group can be advantageously introduced to polymer by guidance to triazine bridge formation, a carboxylic acid, etc. using a cyano basis. It is also possible by being able to use triazine bridge formation of a cyano basis as bridge formation of high performance and a heat-resistant fluororubber, and on the other hand making the cyano basis of a homopolymer or a copolymer into a carboxylic acid to make a polymer into a polyelectrolyte and to use as an electrolyte for cells etc. Moreover, ion bridge formation is also possible. The crosslinked polymer (rubber) which has a cyano basis in a side chain using the ** HF reaction of a principal chain can also be obtained. The polymer which furthermore made the cyanogen machine the carboxylic acid can also be used as ion exchange resin, a high water absorption macromolecule, etc. Moreover, the 3 organic-functions unsaturated compound obtained by the 3 quantification using the cyano basis can be used as a cross linking agent etc.

[0017]

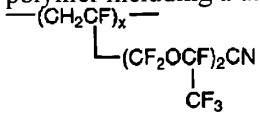
[Example]

Example 1ICH2CF2CF2CF(CF3) CN25g, 15g [of zinc powder], and dimethylformamide (DMF) 10ml was put into the flask, and this was heated on the 160-degree C oil bath. The sudden reaction occurred after a while (after about 40 minutes). Then, mixture was made to flow back at 71 degrees C, and the reaction was continued for 1 hour. After the reaction end, reaction mixture was distilled simply by the ordinary pressure, and 2.43g (99.4% of purity) of products was obtained. The boiling point of 71 degrees C. When the structure of a product was checked by IR, GCMS, and NMR (H, F), it was CH2=CFCF2CF (CF3) CN. IR chart is shown in drawing 1 .

[0018] Example 2ICH2CF2[CF2OCF(CF3)]2CN50g, 20g of zinc powder, and DMF10ml were put into the flask, and this was heated on the 160-degree C oil bath. The sudden reaction occurred after a while (after about 60 minutes). After the reaction end, reaction mixture was made to distill by the ordinary pressure, and the 7.77g fraction was obtained (74% of purity, 11% of unreacted objects, DMF13%). This was refined by simple distillation and the product of 98.6% of purity was obtained. The boiling point of 108-112 degrees C. When the structure of a product was checked by IR, GCMS, and NMR (H, F), it was CH2=CF[CF2OCF(CF3)]2CN. IR chart is shown in drawing 2 .

[0019] Example 3CH2=CF [CF2OCF (CF3)] When 2CN(it is hereafter called "ACN2" for short) 5g and [H(CF2CF2)3COO]2/C2F3Cl3 (8 % of the weight)0.5g were mixed in the glassware, and the nitrogen

purge of the atmosphere in a container was carried out and having been agitated at the room temperature, the viscosity of mixture rose. Reduced pressure distilling off of the low-boiling point object was carried out from reaction mixture, and transparently and colorlessly viscous polymer 2.82g was obtained. It was checked from the result of IR, 1 H-NMR, and 19 F-NMR that generation polymer is polymer including a unsaturated bond. Structure is [Formula 4].



It came out. When calculated from the result of yield and NMR, number average molecular weight was about 35000 - abbreviation 48000.

[0020] Copolymerization with ACN2, CH2=CF2, CF2=CF2, or CF2=CF2/CF3CF2CF2O[CF(CF3) CF2O]2 CF=CF2 (phiVE) was performed like four to example 6 example 3. A result is shown in Table 1.

[0021]

[Table 1]

	モノマー等	仕込量	収量	Mn(収量)	Mn(NMR)	組成(%)(NMR)
実施例4	ACN2 $CH_2=CF_2$ DHP	10g 9Kg/cm²G 1g	6.15g	53,000	43,200	ACN2: 76.9 $CH_2=CF_2$:23.1
実施例5	ACN2 $CF_2=CF_2$ DHP	10g 8Kg/cm²G 1g	6.52g	56,900	1)	ACN2: 78.8 $CF_2=CF_2$:21.2
実施例6	ACN2 φVE[2)] $CF_2=CF_2$ DHP[3)]	1g 20g 5Kg/cm²G 1g	7.99g	68,900	72,300	ACN2: 4.2 φVE: 21.5 $CF_2=CF_2$:74.3

注：1) NMRでは末端基による吸収が小さく、分子量は計算できなかった。

2)φVE：$CF_3CF_2CF_2O[CF(CF_3)CF_2O]_2CF=CF_2$

3)DHP：{$H(CF_2CF_2)_3COO-\}_2$ 8wt%／トリクロロトリフルオロエタン

[0022] To the autoclave of example 7 content-volume 3L, 1225g of pure water, 4122.5 g CF3CF2CF2OCF(CF3) CF2OCF(CF3) COONH, NaCl4g, Na2SO3 2.155g, and 2367.5 g CF3CF2CF2O[CF(CF3) CF2O]2 CF=CF are taught. first the atmosphere in an autoclave with nitrogen Subsequently, it replaced by CF2=CF2, internal pressure was set to 2.5 kg/cm2G by CF2=CF2 at 15 degrees C, and the solution which dissolved 24.5mg of ammonium persulfates in 10ml of pure water was prepared after that. 2211.57 g ICF2CF2CF2CF was taught in the place where the pressure fell to 2.5 kg/cm2G along with advance of a polymerization. The pressure was returned to 2.5 kg/cm2G by CF2=CF2 in the place where the pressure furthermore became 1.5 kg/cm2G.

[0023] Similarly CF2=CF2 was again taught from the bomb in the place to which the polymerization preassure force fell to 1.5 kg/cm2G, and the pressure was returned to 2.5 kg/cm2G. At this time, it considers as the weight of CF2=CF2 which taught weight reduction of a bomb. This procedure was repeated and 2.79g of ACNs2 was taught in the place which the polymerization progressed further and taught a total of 50g of CF2=CF2. Although the rate of polymerization became extremely slow when ACN2 was taught, while carrying out for a while, the rate of polymerization was recovered. Furthermore the polymerization was advanced and 2.79g of ACNs2 was taught once again in the place which taught a total of 100g of CF2=CF2. Although the fall of a rate of polymerization was accepted like last time, it

recovered similarly. The polymerization was suspended in the place which added a total of 140g CF2=CF2. The residual gas monomer was blown and the dispersing element of 1892.2 g was obtained. The solid-content content was 20.87 % of the weight. Polymerization time was a total of 154 minutes. Coagulation of the hydrochloric acid was added and carried out to the obtained dispersing element, the acetone washed, the vacuum drying was carried out at 120 degrees C, and white rubber-like polymer was obtained. Composition of this polymer was CF2=CF2/phiVE/ACN2=76.4/22.8/0.8 (mole ratio). Moreover, Mooney viscosity was ML1+10(100 degrees C) =41.5.

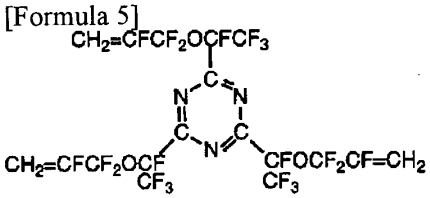
[0024] Rubber-like polymer was obtained like the example 7 except having performed the addition of example 8ACN2, when the sum total charges of CF2=CF2 were 25g and 75g further. When the polymer obtained in the examples 7 and 8 was made into the thin film and IR was measured, the absorption of the 2250cm-1 neighborhood based on -CN was accepted by both. Composition of this polymer was CF2=CF2/phiVE/ACN2=76.1/22.7/1.2 (mole ratio). Moreover, Mooney viscosity was ML1+10(100 degrees C) =40.

[0025] The tetrapod phenyl tin of 3 weight sections was kneaded by the 3 inch rubber covered roll in the polymer 100 weight section obtained in the example 9 example 8, and when press cure was carried out at 230 degrees C for 1 hour, transparent and colorless thin film vulcanized rubber was obtained. With IR of this thin film - The absorption based on CN disappeared and absorption of 1550cm-1 based on a triazine ring was accepted.

[0026] NH315g was taught to example 10CH2=CFCF2OCF(CF3) CN51.5g at -30 degree C, superfluous NH3 was wide opened with atmospheric pressure, the overnight reaction was carried out [a temperature up is gradually carried out to a room temperature,] further, and CH2=CFCF2OCF(CF3) C (NH) =NH was obtained by vacuum distillation. (Structure was checked by IR and NMR.) CH2=CFCF2OCF(CF3) CN23.7g of mols [g / CH2=CFCF2OCF(CF3) C(NH) =NH25.4/ which was obtained] was made to react, and CH2=CFCF2OCF(CF3) C(=NH)-N-C(NH2) CF(CF3) OCF2 CF=CH2 was obtained. (Structure was checked by IR and NMR.) When CH2=CFCF2OCF(CF3) COF5.16g of a double-precision mol was added to this CH2=CFCF2OCF(CF3) C(=NH)-N-C(NH2) CF (CF3) OCF2 CF=CH24.91g, quiet generation of heat was accepted. And although the white solid-state deposited, it became transparent and colorless after a while. Although generation of triazine was accepted from IR of the reaction mixture in this time, it was also checked that the unreacted raw material remains. When triethylamine 3.03g was added to this, it reacted in generation of heat, and liquid was colored a yellowish brown color, and what the raw material reacted completely and the triazine of the following formula generated was accepted from IR (drawing 3).

[0027]

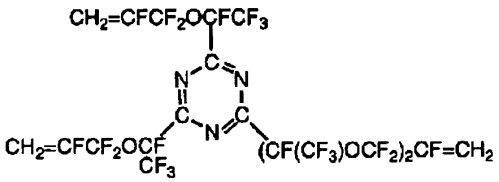
[Formula 5]



[0028] Like example 11 example 10, ACN2 of mols [NH / CH2=CFCF2OCF(CF3) C(NH) =] was made to react, and CH2=CFCF2OCF(CF3) C(=NH)-N-C(NH2) [CF(CF3) OCF2]2 CF=CH2 was obtained. CH2=CFCF2OCF(CF3) COF and the triethylamine of a double-precision mol were added to this, and the following compound was obtained.

[0029]

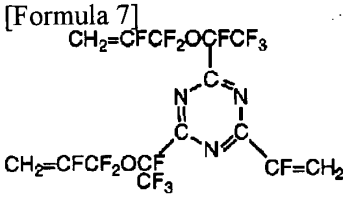
[Formula 6]



CH2=CFCOF and the triethylamine of a double-precision mol were added to CH2=CFCF2OCF(CF3) C (=NH)-N-C(NH2) CF(CF3) OCF2 CF=CH2 obtained like example 12 example 10, and the following compound was obtained.

[0030]

[Formula 7]



[0031] It is organic peroxide (the par hexa -2, 5-B.) to [CH2=CFCF2OCF (CF3) triazine CN] 31.0g obtained in the example 13 example 10. When 0.1by Nippon Oil & Fats Co., Ltd. g was added and it heated at 160 degrees C on the oil bath, it reacted rapidly and the stiff resin-like object was obtained.

[0032] When triazine 0.5g and triaryl isocyanate (TAIC) 0.5g obtained in the example 10 were mixed like example 14 example 13, the par hexa -2 and 5-B0.1g were added to this and it heated at 160 degrees C on the oil bath, it reacted rapidly and the stiff resin-like object was obtained.

[0033] Copolymer rubber of the tetrafluoroethylene which has iodine in example 15 and example of comparison 1 both ends, and CF3CF2CF2O[CF(CF3) CF2O]2 CF=CF2 (77 mol % of tetrafluoroethylene contents.) The skin-reactive-factor(carbon filler) 7 weight section, the MTC(carbon filler) 8 weight section, the triazine CH2=CFCF2OCF(CF3) CN8.5 weight section obtained in the example 1 and the par hexa -2, and the 5-B-2 section are kneaded by the 3 inch rubber covered roll in the Mooney viscosity ML(1+100) (100 degrees C) =26 100 weight section of a copolymer, and it is JSR. CURELASTOMETER The vulcanization curve was measured at 160 degrees C using IIF The component was kneaded like the above except carrying out TAIC3 weight section combination instead of CH2=CFCF2OCF(CF3) CN as comparison.

[0034] Press cure of the kneading object was carried out for 10 minutes at 160 degrees C, respectively, the sheet with a thickness of 2mm was obtained, and it vulcanized secondarily at 200 degrees C for 4 hours. About the ordinary state physical properties (at the time of fracture intensity and elongation) of a vulcanization sheet, it is ORIENTEC. TENSILON is used and it is JIS. K It measured according to 6301. After making the vulcanization sheet manufactured similarly heat-age in 250-degree C oven for 70 hours, aging physical properties were measured. A result is shown in Table 2.

[0035]

[Table 2]

	実施例1.5	比較例1
常態物性		
破断時強度(kgf/cm²)	86	120
破断時伸び (%)	136	100
老化物性		
破断時強度(kgf/cm²) (変化率)	64 (−26%)	79 (−34%)
破断伸び (%) (変化率)	170 (+26%)	205 (+105%)

[Translation done.]

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 10-237130

(43)Date of publication of application : 08.09.1998

(51)Int.Cl. C08F114/18
C07C255/13
C07D251/24
C08F290/06
C08F299/02

(21)Application number : 09-037489

(22)Date of filing : 21.02.1997

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(54) FLUORINE-CONTAINING NITRILE AND ITS POLYMER

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain a new compound obtainable in a relatively good yield and having good storage stability and high polymerization activity.

SOLUTION: This compound is represented by the formula CH2=CFCF2O-(CF2 O)x-(CF2CF2O)y-(CX12CF2CF2O)z-(CFX2CF2O)w-CFX3-CN [X1 is H, F or Cl; X2 is H or Cl (trifluoro)methyl; X3 is H, F, Cl or trifluoromethyl; (x), (y), (z) and (w) are each 0-20 and the sum of (x), (y), (z) and (w) does not exceed 20], e.g. a compound of the formula CH2=CFCF2O-CF(CF3)-CN. The compound of the formula CH2=CFCF2O-(CF2O)x-(CF2CF2O)y-(CX12CF2CF2O)z)-(CFX2CF2O)w-CFX3- CN is obtained by removing FI from terminal iodine nitrile of the formula ICH2CF2CF2O-(CF2O)x-(CF2CF2O)y-(CX12CF2CF2O) z-(CFX2CF2O)w-CFX3-CN in a solvent (e.g. dimethyl-formamide) in the presence of a catalyst (e.g. zinc) at a temp. of -20 to 200°C.

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[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]